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Information Update

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Update # 37

1. The Arizona revised Administrative Rules for the environmental Laboratory Licensure Program have been approved by the GRRC (Governors Regulatory Review Council) and will be submitted to the Secretary of State's office for promulgation. Upon promulgation the rules will be implemented.

2. As per a communication with EPA MICE (Methods Information Communication and Exchange, Tel: 703-821-4690), SW 846 Update III is being mailed out in June to all the subscribers and that the newer methods will be implemented by EPA once they are mailed out. Unlike drinking water methods no transition period is given for the implementation of the newer methods for the solid waste.

3. Recommendation from MICE regarding setting laboratory's matrix spike limits for 8000 series methods. Laboratories can set their own limits based on their historical data. Their recommendation was to set individual limits for different matrices; For e.g., TCLP, low level waste, high level waste, oil and so on.

4. Since the EPA method 608 is not specific enough for the multi-peak components (PCBs, chlordane and toxaphene), Laboratory Licensure Program has set the following QC criteria for the method:

    a. **Initial Calibration:** For each single-component pesticide, a three level calibration curve must be established. Initially, only one Aroclor is required to have a full calibration curve , however, all the other multi-peak components must be shot at the laboratory reporting level. In addition, if any of the multi-peak components is detected in any sample, then the analyst must run a full curve for that analyte detected.

    b. **Calibration Verification:** For each single-component pesticide, run one of the standards and quantitate. Run the mid-point aroclor standard used for the full initial curve and quantitate. Run a toxaphene and chlordane standard at any level, for pattern recognition.

    c. **QC Check and/or Matrix Spike:** Spike at any level for each single-component pesticide and any one of the multi-peak components, that can be quantitated.

5. It is not required for all the replicates to be done on one day for Method Detection Limit Studies.

However, the selection of the replicates should not be based on obtaining the lowest MDL value. For a data point to be discarded, it must be a statistical outlier and the laboratory's protocol must be documented. MDL spike level should not be more than 10X greater than the resulting MDL. It is best to follow the 40 CFR suggestion of running 2 aliquots and checking them first for appropriate spiking level. If they are acceptable then only 5 other aliquots are required. The laboratories could also base the spiking levels on the previous year's MDL study results.

For 608, 8080 and 8081, the MDL study for each parameter (single component-pesticides, aroclors, chlordane and toxaphene) must be established. Although a method detection limit is a useful benchmark for evaluating and comparing method sensitivity, it may not be an appropriate indicator of the level at which a multi-peak compound can be identified. Laboratory Reporting Limits established should be able to identify the peak patterns for multi-peak compounds.

6. Questions have been raised by several laboratories regarding the requirements for the initial method capability study if a method is being set up for a partial compound list. Arizona Department of Health Services has several concerns about method validation for only a partial list of target analytes.

    a. Coelution, partial or complete, is a problem if a laboratory analyzes only a few compounds out of a much larger list. If the laboratory never analyzes the other compounds in a standard or sample and they optimize conditions for just the few analytes of concern, and if the other analytes in the EPA list are in the sample, they could have coelution problems of which they are not aware.

    b. For dual column analyses (i.e. PCBs/Pesticides), a laboratory must be careful not to shorten the run time so that even a second column would not adequately separate the possible coeluters.

    c. For GC/MS, coelution is an issue if the same quantitation ion is used for the coeluting compounds or if the secondary ion of a coeluter is used as the quantitation ion of the other coeluter (i.e. Indeno[1,2,3-cd]pyrene and Dibenz[a,h]anthracene). Also, a laboratory must be careful not to shorten the run time to the point where column overloading occurs or many compounds may be coeluting and clean spectra cannot be obtained for each compound, causing identification problems.

    To address the above concerns, while taking into consideration the needs of the laboratory community, the Office of Laboratory Licensure has developed the following criteria for a partial list and or a complete list method validation.

    a. Due to the mass selectivity of the detector, our crireria is that a laboratory may perform partial list method validation for GC/MS or LC/MS methods as long as different ions are used for quantitation of coeluters. The laboratory must also ensure that strong secondary ions of a coeluting peak are not being used as the quantitation ion for the other coeluting peak .

    b. For dual column analyses (i.e. pesticides and PCBs), as long as a laboratory can demonstrate that under routine operating conditions all components are separated between the two columns, it is acceptable not to show full list capability.

    c. For single column analyses, the laboratory must use a second dissimilar column or GC/MS confirmation. The laboratory must also demonstrate that under routine operating conditions, all components are separated between the two columns. It is then acceptable not to show full list

capability.

d. Confirmation is required for all reportable compounds if a single column GC analysis was performed, if historical data of the confirmation of the compounds are not available. Qualitative confirmation must be performed on one sample per site in order to characterize that site with the presence of specific contaminants. If the project continues, the previously performed confirmation analyses remains valid. If a new compound is found in the site, it must be confirmed. If the laboratory does not want to comply with the confirmatory requirements, as stated in the methods, the laboratory must qualify test reports by indicating that GC provides a tentative identification. A foot note will be required to be included addressing the confirmation requirements. The footnote should state whether the identification is tentative and needs confirmation or the identification has previously been confirmed. Please note that this qualifier still requires that the method QA/QC criteria are met. This footnote is not to be used as an excuse for poor chromatography or sloppy data interpretation when the method will yield enough information for reasonable data interpretation accuracy. Be advised that the Arizona Department of Environmental Quality (ADEQ) may or may not accept data for compliance purposes with the said qualifier. The Office of Laboratory Licensure strongly urges the laboratories to check with the client and or ADEQ before adopting this procedure.

e. It is the laboratory's responsibility to ensure that the above concerns have been adequately addressed for either single or dual column analyses. If during the course of a survey ADHS has further concerns about the identification and quantification of analytes, the laboratory will be required to perform a Proficiency Evaluation (PE) sample designed by ADHS that would demonstrate that analytes are being accurately identified and quantified (These results would indicate whether further method development or GC/MS confirmation is required).

7. Our office requires that laboratories notify the clients in the final report when a dilution was required for sample analyses. The reporting limits should get multiplied by the dilution factor. If the MDL is a statistical number, it does not get multiplied but if it is a quantifiable number included in the curve, then it should get multiplied.

8. If you have any questions regarding the Updates, or if you have any technical questions that need clarification, please call or send e-mail to Prabha Acharya, Program Manager, Technical Resources and Training at the Laboratory Licensure. A table of contents to all the Information Updates published is also available.